First Quarterly Status Report

on the
Role of Anions in Mechanical Failure

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ABSTRACT

To date all effort has been expended on phase 1 of the research program, effect of anions on fabrication of MgO. Materials have been obtained, a hot press is operating and approximately 50 pressings have been conducted. Chemical analyses of these are underway.

INTRODUCTION

As stated in the initial proposal, the primary purpose of this research is to investigate the effect of anion impurities on the behavior of polycrystalline ceramics, especially the mechanical behavior. A variety of likely anion impurities are to be used including F^-, $S^=$, Cl^-, OH^-, and OD^-. Four specific effects of these anion impurities are to be determined. These are:

1. Effect of the anions on the fabrication (by hot pressing) of MgO.
2. Effect of anions on grain growth.
3. Effect of anions on grain boundary diffusion of substitutional cations.
4. Effect of anions on grain boundary microhardness.

The last of these investigations, the microhardness and diffusion must sequentially follow the first two since the specimens produced in the fabrication study will be used for grain growth studies and then subsequently for the final two investigations. Consequently, all of the effort at this time has concentration on the production of appropriate hot pressed pieces of magnesium oxide containing the appropriate dopants, and confirmation of the presence of the impurity by chemical analysis.

MATERIALS

The materials required for the fabrication of test pieces in these investigations have been ordered and in most cases have been received. They are listed in Table 1.

EQUIPMENT

Since a suitable hot pressing faculty was obtained from Jet Propulsion Laboratory, extensive construction and development of a new test faculty was not required. In addition, the unit obtained is far superior in total capability than would have been available within the limits of this program. This press was successfully received with only minor damage, has been set up and is now working satisfactorily. It has been more extensively described elsewhere.[1]

The grain growth studies and reheats which have been conducted to date have made use of a conventional silicon carbide resistance furnace having

an aluminum oxide muffle tube. It has been used with an air atmosphere to date.

PROCEDURE

The preparation of magnesium oxide powders doped with anion impurities, involved weighing and mixing by means of a V blender in 75 gram lots. Several 1/2 gram samples were then analyzed from the mixed lot to confirm the desired amount. Reported results are analytical values not preparation values. In general, these agreed within five per cent. The analytical procedures used to date have been sulfur by combustion and iodimetric titration and chlorine by precipitation.

The loading procedures for the hot pressing dies consisted of weighing, cold compacting, and placing in the press. A variety of powder pre-treatments have been used to date such as calcining in vacuum, absolute alcohol wash, and storage in the presence of desiccant. To date these techniques have not been standardized and so will not be recorded in detail until a final selection is made.

From previous studies of hot pressing magnesium oxide, it has been found that if compaction occurs too early in the thermal cycle, excess gas is entrapped within the powder and end density cannot be obtained. If the pressure is applied too late, the reactivity of the fine particle powders is reduced and complete compaction again does not occur. For this reason, the procedure used here involves a linear increase in temperature from approximately 300 ^{o}C to maximum with a concurrent increase in pressure to obtain maximum pressure shortly before maximum temperature. The compact is then held at maximum temperature and pressure until no further compaction is evident. The system is capable of detecting compaction motion as small as .0001 ins. The pressure is then released, the heater power turned off, and the specimen is allowed to furnace cool.

RESULTS

To date almost 50 specimens have been pressed as the pressing parameters for the obtainment of full density are determined. Some re-

presentive results are shown in Table II. To date no meaningful conclusions concerning the effect of anions on hot pressing have been obtained. Work will continue on the development of the fabrication parameters and reheat studies to determine the effect of the anions on grain growth are expected to begin shortly.

REFERENCES

1. Martin H. Leipold and Thomas H. Nielsen, "Hot Pressed High Purity Polycrystalline MgO," Am. Cer. Soc. Bull. 45 (3) 281 (1966).

Table I. Materials Used for Fabrication of Anion Doped Hot Pressed MgO

MgO	Fisher M-300 Electronic Grade Lot 771932
MgF_2	Vactran British Drug Houses Ltd. BDH Laboratory Chemical Division Poole Dorset 827156/690701 (200 mesh)
S	Sulfur Flowers Powder Will Scientific, Inc. W1 86638
$MgCl_2$	Baker & Adamson Allied Chemical Lot A328
$Mg(OH)_2$	Kanto Chemical Works Tokyo, Japan
MgO	Baker Chemical Co. Lot #31792
D_2O	Isomet Corporation 99.9%

Table II. Typical Hot Pressing Parameters for MgO Containing Anion Dopants

Pressing #	MgO Type	Dopant	Pressing Parameters T °C	P KSI	Atm	Density gm/cm^3
6	Baker	None	1120	17.5	V†	3.46
21	Fisher	None	960	22.0	V	3.44
27	Fisher	None	960	22.0	V	3.52
32	Fisher	0.6 a%S	960	22.0	V	3.52
41	Fisher	2.9 a%S	960	14.0	Argon	3.20
44	Fisher	F*	965	14.0	Argon	3.26
47	Fisher	3.34a% Cl	1160	17.5	V	3.54

† Mechanical Pump Vacuum ≈200 μ.

* Analysis not yet available